

Spectral Analysis of Reflected Soft X-ray for Detecting Foreign Materials in Foods

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A continuous soft X-ray system with a semi-conductor detector was used to detect foreign materials in food samples. Basic properties of transmitted and reflected soft X-ray for some metallic and non-metallic materials were measured using the Si-PIN sensor equipped with a multi-channel analyzer. Soft X-ray spectra in photon energy were obtained for copper, galvanized iron, acrylic, vinyl chloride and glass of different thickness as foreign materials in foods. This detecting system was capable of analyzing intensities of transmitted and reflected X-rays at each energy level in the broadband. The reflected soft X-ray spectra were found to have characteristic spectrum curves for each sample. In order to detect acrylic, vinyl chloride and glass in a hamburger patty, the reflected soft X-ray spectra were analyzed and this technique was found to have the potential for more sensitive detection of foreign materials. The detecting system using properties of transmitted and reflected X-rays at each energy level is considered to be valid for non-metallic foreign materials.

Keywords: soft X-ray, spectra, transmittance, reflectance, detection, physical foreign material, food

In general, X-rays are used for non-destructive inspections in the medical and industrial fields owing to their transmissibility. In the field of food and agricultural process engineering, X-rays have been used to detect foreign materials in foods as a non-destructive technique for food quality control (Kenmochi, 1992; Zwiggelaar *et al.*, 1996). Current inspection methods for detecting physical foreign materials are based on characteristics of a transmitted X-ray image through a material. The pixel intensity of the transmitted X-ray image through an object is determined by its thickness and absorption coefficient μ , which is based on Beer's Law and varies with constituent elements and bulk density. Although the chemical component of a material can be determined using X-ray absorption fine structure (XAFS) techniques, which measure μ value with high resolution (Grieken & Markowicz, 1993), the performance of the transmitted X-rays system is much poorer in commercial use for the industrial food field. Furthermore, current X-ray inspection devices are not as valid for food and agricultural materials because of their non-homogeneity and varying thickness (Morita *et al.*, 1996).

X-rays can transmit through or reflect from a material. The reflected X-rays consist primarily of fluorescent and scattering components (Kikuta, 1992). Scattering X-rays from the surface of a material can provide the characteristic properties of the material, especially of a metallic material (Bossi *et al.*, 1988; Schatzki & Wong, 1989). Food materials consisting of light elements have minimal μ value and X-rays are transmitted through them more than through metallic materials. Therefore, reflected X-rays from food materials would also include sub-surface characteristics.

In this preliminary study, soft X-ray transmission and reflection spectra were measured and analyzed for non-metallic mate-

rials to gain understanding of the basic principles of detection of foreign materials in foods.

Detecting foreign materials with soft X-rays X-rays are electromagnetic waves similar to visible and NIR (near infrared) radiations. Laws of reflection and refraction in the visible and NIR regions are well known and have been used extensively for quality evaluation. However due to their shorter wavelengths, X-ray radiations have strong transmittance property as compared to visible and NIR radiations. The region of wavelengths for X-rays is from 10 nm to 0.01 nm and is usually expressed as energy levels from 0.12 keV to 120 keV (Kikuta, 1992). The higher the energy is, the stronger the transmission through materials. For materials with smaller atomic numbers, or lower bulk densities, or less thickness, the intensity of X-ray transmitted through the material is higher. As the energy of soft X-ray is low, its transmission power is weak. Therefore, soft X-ray is rarely used in medical or industrial fields. However, as the wavelength region of soft X-ray is on the same order as an atomic diameter, the intensity of X-ray transmitted through the material changes with even a slightly different density of the material.

The intensity of continuous soft X-rays is usually expressed by a spectrum curve. This spectrum contains a higher percentage of X-rays in a low energy region. Therefore, soft X-ray measurement can indicate a small change in small density in a material with high sensitivity.

Materials and Methods

Soft X-ray source and detector A specially modified device, model TRIX-60 (TOREX Co., Ltd) was used as a soft X-ray generator in this study. It can generate variable power levels and includes an inverter circuit for power stabilization. The soft X-ray tube (model H-7131 from Hitachi Co.) uses a tungsten cathode and a Beryllium radiation window. The beryllium win-

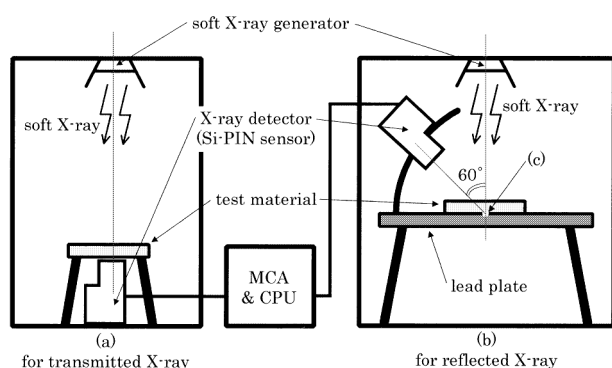


Fig. 1. Schematic diagram of measuring apparatus.

down absorbed some but not all of the soft X-rays generated. Under the operating conditions of less than 60 kV in voltage and 10 mA electric current of the soft X-ray tube, a continuous soft X-ray beam with energy levels from 6 to 30 keV was obtained. The peak of the energy spectrum was at approximately 8.5 keV, which was in the soft X-ray region.

A Si-PIN sensor and a multi-channel analyzer were used in this study as the X-ray detecting system. The photons detected by the Si-PIN sensor (model XR-100T, from AMPTEK Inc.) were transformed into an electrical signal, which was further enhanced by an amplifier (model PX2T from AMPTEK Inc.) and then digitized into 2048 discrete energy levels by a multi-channel analyzer (model S5000 from APTEC Eng. Ltd.). The data acquisition and analysis were done on a personal computer. The FWHM (full width at half maximum level) for an Americium-241 standard was calibrated at 308 eV in this system. The spectral energy resolution was 15.4 eV in our experiments.

Measurement techniques Acrylic ($[-CH_2-CH(CN)-]_n$), glass (SiO_4) and vinyl chloride ($[-CH_2-CHCl-]_n$) were used in this study as foreign materials. They were apparently transparent in the visible range of the electromagnetic spectrum. A hamburger patty was also tested with and without these foreign materials.

A diagram of the experimental apparatus for both soft X-ray transmittance and reflectance tests is shown in Fig. 1. For the measurement of transmitted X-rays, the Si-PIN sensor was positioned exactly beneath the X-ray tube using a diode laser (Fig. 1-(a)). The test materials were positioned on top of the detector for all transmittance measurements. The thickness was 0.65 mm for glass, 0.50 mm for vinyl chloride, and 2 mm for acrylic.

To measure reflected X-rays, the Si-PIN sensor was positioned above the test materials (Fig. 1-(b)). The distance between the X-ray source and point (c) was fixed at 450 mm, and the distance between the detector and point (c) was fixed at 300 mm for all reflectance measurements. The detecting angle of reflection was held at 60 degrees from the vertical plane. A lead plate 2 mm thick was used as background material, and it was positioned horizontally to the incident X-ray beam. The test materials were positioned on the lead plate for all reflectance measurements. All test materials were 2 mm thick, and were stacked to provide 4, 6 and 8 mm thicknesses.

A set of three soft X-ray spectra was measured for each test material. Each spectrum was smoothed using the Savitzky-Golay method with a 9-point window (Gorry, 1990). The smoothed

spectrum was then averaged for each set of the measurements.

Results and Discussion

Basic spectrum of generated soft X-rays The spectral curve of generated soft X-rays, detected at a distance of 300 mm between the soft X-ray source and the detector, is shown in Fig. 2. The soft X-ray power settings were set at 20 kV and 0.5 mA. The numbers of detected photons per mm^2 per s were counted at each photon energy level in keV and the spectral curve of the soft X-ray was plotted from 6 keV to 30 keV. The spectrum showed 3 peaks at 8.3, 9.6 and 11.2 keV, these represented the characteristics of the tungsten cathode in the soft X-ray tube. Soft X-rays of less than 6 keV in energy level were mostly absorbed by ambient air; those with such lower energy levels are close to the wavelength of ultraviolet light regions. In general, there are two X-ray regions: one is a soft X-ray region and the other is a hard X-ray region. However, there is no clear definition between soft X-ray and hard X-ray in energy level. As the X-ray region from 6 keV to 30 keV is considered to be a low energy level, it is also referred to as the soft X-ray region in this study.

Measurements of transmitted soft X-rays Spectra of transmitted X-rays through acrylic, vinyl chloride and glass materials with different thickness are shown in Fig. 3. Although the intensities of transmitted X-rays were changed for each material, the peak energy positions for each spectral curve were similar to the basic spectrum of generated soft X-ray. The peak positions for acrylic and vinyl chloride at the 8.3 keV peak region showed a slight shift to higher energy levels due to beam hardening compared to

$$\mu = -\frac{1}{x} \ln \left(\frac{I}{I_0} \right), \quad (1)$$

where,

I_0 : incident X-ray intensity (photons/ $mm^2 \cdot s$)

I : transmitted X-ray intensity (photons/ $mm^2 \cdot s$)

x : thickness of tested material (mm)

the basic spectrum generated soft X-ray. The transmitted soft X-ray of the glass was mostly absorbed due to its high density. In the soft X-ray transmittance properties, these spectral curves for the test materials contained both properties of thickness and density. Therefore, soft X-ray linear absorption coefficients (μ) for

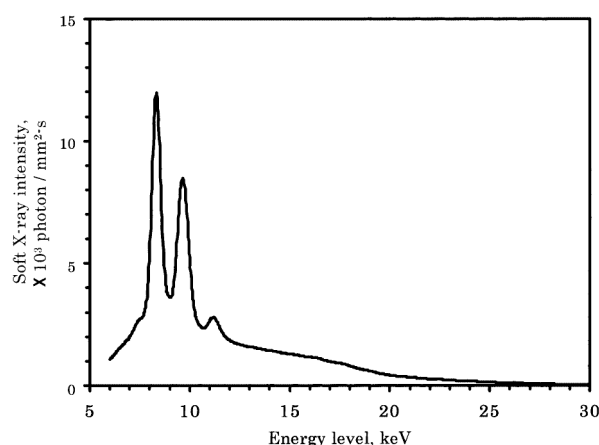


Fig. 2. Spectral curve of generated soft X-ray at 300 mm from the source. (generated power: 20 kV, 0.5 mA)

each material were calculated at each energy level, according to the following Beer's Law equation (1) (Zevin & Kimmel, 1995).

Figure 4 shows the calculated μ values in the region from 7 keV to 17 keV for these test materials. The μ values for each material decreased exponentially with increased photon energy levels. As the μ values is higher with high density of a material in the region of low photon energy, the μ values of test materials were in the sequence of glass, vinyl chloride and acrylic. Furthermore, the μ values were more variable in the region from 7 keV to 12 keV in this study. The soft X-rays are expected to be transmitted more easily in acrylic than glass and vinyl chloride due to its lower μ value. Although the soft X-ray transmittance characteristics of a material can be determined by the changes in μ value within a small energy level region in the XAFS method, this method requires knowledge of the material thickness before measurement. It therefore is not appropriate for food and agricultural products that have different forms and sizes and non-homogeneous structures.

Measurements of reflected soft X-rays Spectral curves of reflected soft X-rays for copper plate at the thickness of 0.10 mm and galvanized iron plate at the thickness of 0.35 mm which

were positioned horizontally to the incident X-ray beam are shown in Fig. 5. The soft X-ray source power setting was at 40 kV and 1.0 mA. The spectral intensities of reflected soft X-rays were obviously much smaller than those of the transmitted soft X-ray in Fig. 3. However, special characteristic peaks in each spectrum were measured at 7.9 and 8.8 keV for copper plate and 6.5, 7.0, 8.5 and 9.4 keV for galvanized iron plate. Figure 6 shows spectral curves of reflected soft X-rays for the lead plate as background at different soft X-ray power levels. Although the spectral intensities were changed depending on the conditions of different soft X-ray source power levels, the spectral curves were the same. The main peaks were positioned at 8.4, 10.5, 12.6 and 14.7 keV. Compared with the spectral intensities of the lead plate in Fig. 6, spectral curves of the copper plate and the galvanized iron plate in Fig. 5 were completely different in peak position and curve shape.

As the μ values of metallic materials with large atomic numbers such as lead are high, soft X-rays cannot transmit through heavy metallic materials. The reflected soft X-ray for these materials can thus contain only information on the material surface, so that the spectral curves showed the characteristic changes of

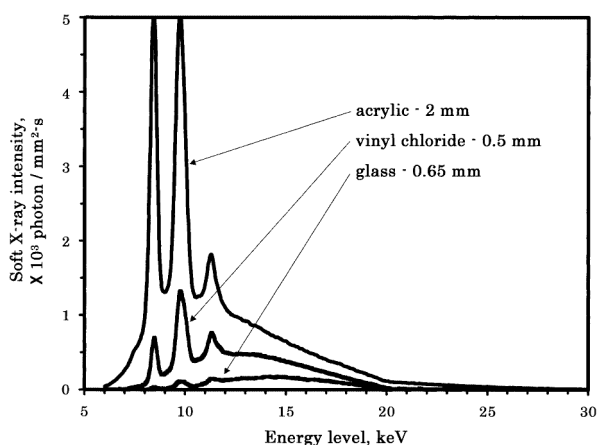


Fig. 3. Spectral curves of transmitted soft X-ray through non-metallic materials.

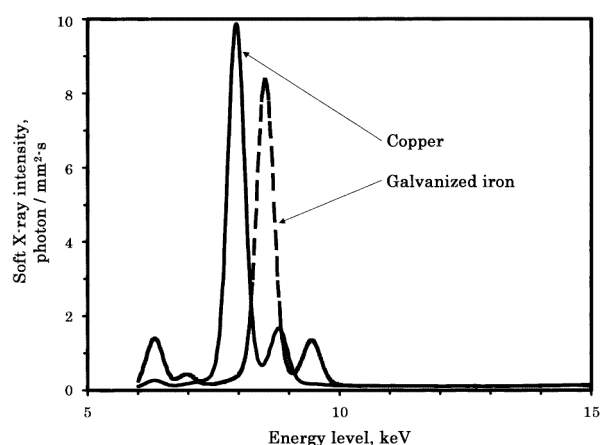


Fig. 5. Spectral curves of reflected soft X-ray for two kinds of metallic materials.

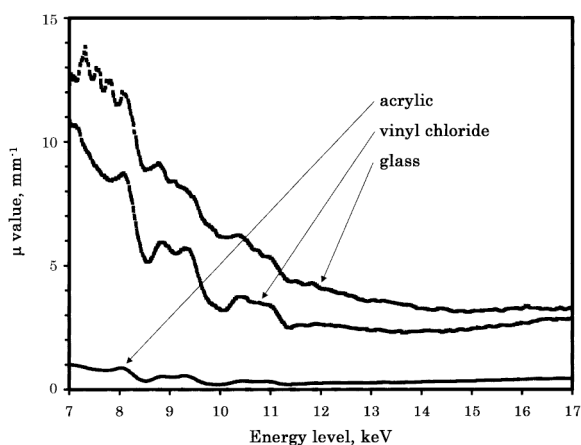


Fig. 4. Changes of μ values at different energy levels for non-metallic materials.

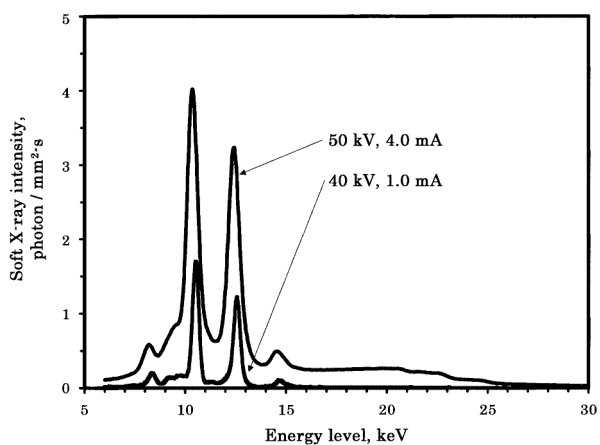


Fig. 6. Spectral curves of reflected soft X-ray for a lead plate at different power levels.

these materials. Figure 7 shows spectral curves of reflected soft X-rays for acrylic plates with different thickness. These plates were placed on top of a lead plate; their spectral peaks were at 8.2, 9.4, 10.5, 12.5 and 14.7 keV, the three latter peaks being in similar peak positions as the lead plate. The two other peaks at 8.2 and 9.4 keV were characteristic peaks for non-metallic materials with a light atomic number. The peak intensities of 10.5 and 12.5 keV were decreased with increased acrylic thickness. To investigate the difference of peak intensities, the peaks at 10.5 and 12.5 keV were magnified and are shown in Fig. 7. The incident soft X-ray was transmitted through the acrylic plate and then reflected at the lead plate, and then the reflected soft X-ray was re-transmitted through the acrylic plate. Therefore, the intensity of reflected soft X-ray was changed according to thickness of the acrylic plate. These peaks were characteristic changes for the acrylic material and distinction of the acrylic material and prediction of its thickness could be estimated by an analysis of these five peak changes.

Figure 8 shows spectral curves of vinyl chloride plates with different thickness; these plates were also placed on top of the lead plate. The spectral peaks of the vinyl chloride plate were at 8.2, 9.4, 11.2 and 25.0 keV and there was no peak for the lead

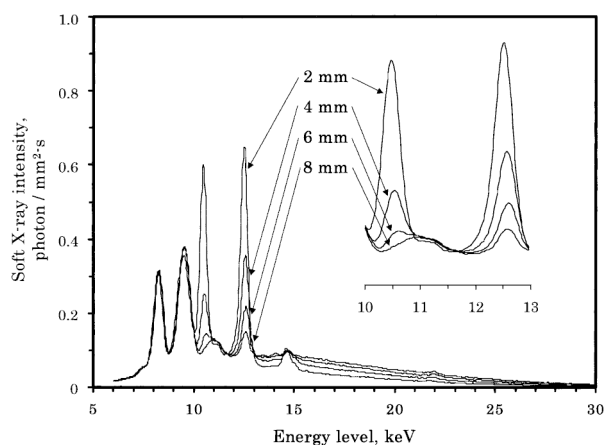


Fig. 7. Spectral curves of reflected soft X-ray from an acrylic plate of different thicknesses.

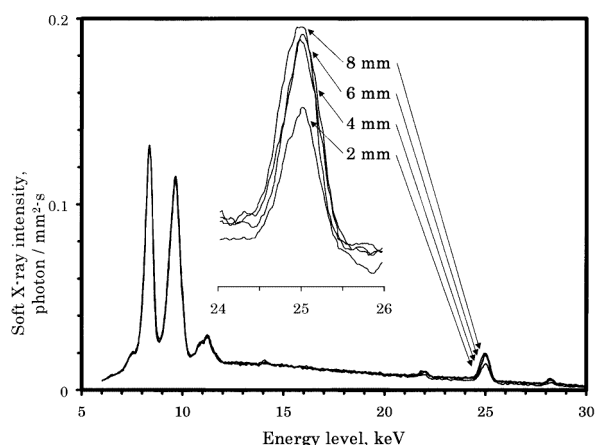


Fig. 8. Spectral curves of reflected soft X-ray from a vinyl chloride plate with different thicknesses.

plate as the background of the μ values for vinyl chloride were higher than for acrylic. In other words, the peaks for lead appeared in the acrylic because of the smaller μ values of acrylic and did not appear in the vinyl chloride due to its higher μ values. These peaks were characteristic peaks for vinyl chloride. The peak intensity of 25.0 keV was increased with increasing thickness of vinyl chloride: this peak at 25.0 keV was magnified and is shown in Fig. 8. The peak of 25.0 keV is caused by an atom of chlorine. In the vinyl chloride material, distinction of the material and its thickness can be estimated by the peak intensity of reflected soft X-ray at 25.0 keV.

Figure 9 shows a schematic diagram of reflected soft X-ray for materials with different densities. In the acrylic material, as the density is light, the incident soft X-ray beam easily reached the lead plate, while in vinyl chloride, as the density is heavier than acrylic density, the incident soft X-ray beam weakly reaches the lead plate. Therefore, the spectral curves for reflected soft X-ray were complete different between acrylic and vinyl chloride for effect of the lead plate.

The spectral curves of reflected X-rays for an acrylic at the thickness of 4 mm, a vinyl chloride the same thickness and a composite material of 2 mm vinyl chloride plate layered with 2 mm acrylic are shown in Fig. 10. The soft X-ray power source setting was at 50 kV and 4.0 mA. Although the peaks at 8.2 and 9.4 keV appeared in all spectral curves, the peaks at 10.4 and

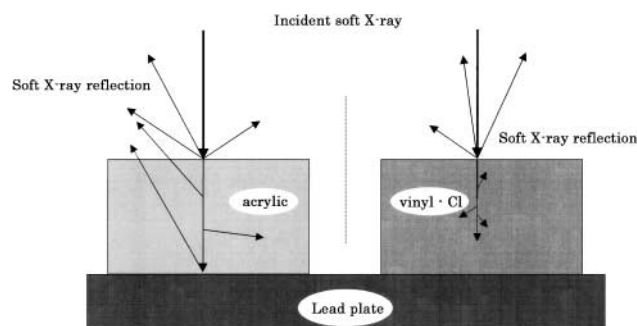


Fig. 9. Schematic diagram of reflected soft X-ray for materials with different densities.

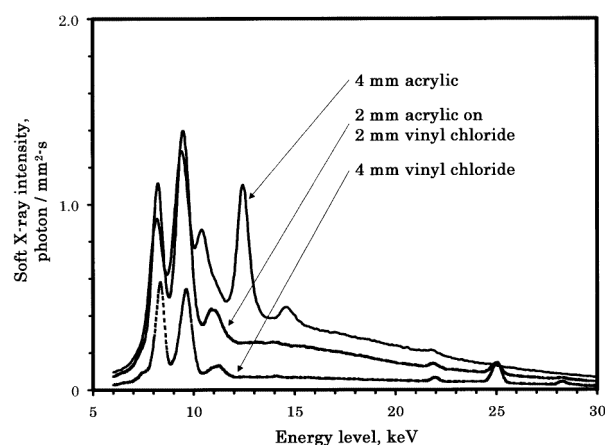


Fig. 10. Spectral curves of reflected X-rays for acrylic and vinyl chloride 4 mm thick and a composite material of 2 mm vinyl chloride plate layered with 2 mm acrylic.

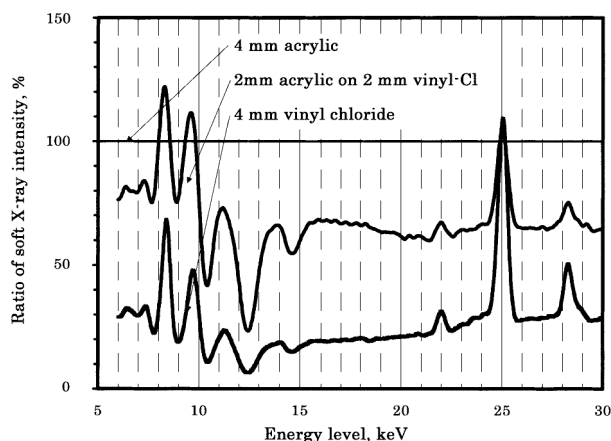


Fig. 11. Ratio of spectral curves for acrylic, vinyl chloride and composite material based on the reflected soft X-ray of acrylic.

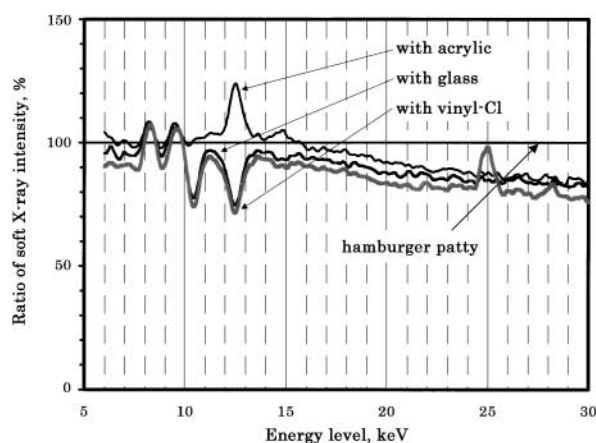


Fig. 12. Ratio curves of reflected soft X-rays for a hamburger patty embedded with acrylic, glass or vinyl chloride as a foreign material.

12.4 keV only appeared for acrylic material, and the peaks at 11.2 and 25.0 keV only for vinyl chloride material. For the composite material, the characteristic peak appeared at 10.8 keV, and its spectral intensity was largely influenced by the vinyl chloride. Basically, the reflected soft X-ray for the lead plate has peaks at 10.4, 12.5 and 14.7 keV and these peaks only remained in the acrylic test. The peak at 25.0 keV was a characteristic peak for vinyl chloride. The spectral curve for the composite material was influenced by both acrylic and vinyl chloride and it showed both properties.

To emphasize these spectral curves, the ratio of spectral curves for acrylic, vinyl chloride and composite material based on the reflected soft X-ray of acrylic were calculated. The calculated spectral curves are shown in Fig. 11. In the composite material, two emphasized peaks were obtained: one peak at 25.0 keV showed the existence of vinyl chloride and the other at 12.4 keV showed the existence of acrylic in the composite material. Consequently, it was clear that the measurement using the spectral curve of reflected soft X-ray could be valid for distinction between these materials.

Figure 12 shows the ratio curves of reflected soft X-rays for a hamburger patty embedded with acrylic, glass or vinyl chloride as a foreign material. The patty thickness was adjusted to 10 mm and the thickness of these foreign materials was 2 mm. In this measurement, the foreign material was embedded in the hamburger patty and the total thickness was kept at 10 mm. The patty with the foreign material was placed on the lead plate with the foreign material face down on the lead plate. The soft X-ray source power settings were at 50 kV and 4.0 mA, and the ratio of spectral curves for the hamburger patty with each embedded material was calculated, based on the reflected soft X-ray of the patty without a foreign material. As the reflected soft X-ray of the foreign material had to pass through the patty, the ratio curve was slightly changed in relation to Fig. 11. Again, two primary peaks were obtained: one at 25.0 keV showed the existence of vinyl chloride and the other at 12.4 keV showed the existence of acrylic in the spectral curves. Thus, the ratio curves could possibly be used to detect a foreign material in a hamburger patty.

Consequently, these findings confirmed that measurement of soft X-ray reflectance properties may have potential to detect for-

eign materials in food.

Conclusions

With the goal of detecting foreign materials in food, soft transmitted and reflected soft X-ray spectral properties for three non-metallic materials, acrylic, glass and vinyl chloride, were measured and their detection in a hamburger patty was also attempted using spectral reflected soft X-ray properties. The spectral data were collected and analyzed using a Si-PIN sensor with a multi-channel analyzer and the results were as follows:

1. The basic spectrum of generated soft X-rays was measured and the spectrum showed 3 peaks at 8.3, 9.6 and 11.2 keV. Spectra of transmitted X-rays through acrylic, vinyl chloride and glass materials with different thickness were measured, and soft X-ray linear absorption coefficients (μ) for each material were calculated at each energy level. Although the μ values were more variable in the region from 7 keV to 12 keV in this study, it was found that the transmitted soft X-ray method for detection of foreign materials was not suitable for food and agricultural products that have different forms and sizes and non-homogeneous structures.
2. Spectral curves of reflected soft X-rays for copper plate and galvanized iron plate as metallic materials were completely different in peak position and curve shape of the basic spectrum of generated soft X-rays.
3. The spectral curves of reflected soft X-rays for acrylic plates with different thickness were measured and two peaks at 8.2 and 9.4 keV were characteristic for non-metallic materials with light atomic number. The peak intensities of 12.5 and 14.7 keV were decreased with increased acrylic thickness. These peaks were characteristic changes for the acrylic material and distinction of the acrylic material and prediction of its thickness could be estimated by an analysis of these peak changes.
4. Combination with the peak intensities at 8.2 and 9.4 keV and the peak intensity at 14.7 keV could be estimated to be acrylic material and its thickness.
5. The spectral curves of vinyl chloride plates with different thickness were also measured and distinction of the material and its thickness could be estimated by the peak intensity of reflected soft X-ray at 25.0 keV.

6. The spectral curves of reflected X-rays for a composite material of 2 mm vinyl chloride plate layered with 2 mm acrylic were measured and calculated, and showed both acrylic and vinyl chloride properties.

7. The ratio curves of reflected soft X-rays for a hamburger patty embedded with acrylic, glass or vinyl chloride as a foreign material were calculated and these foreign materials showed characteristic peaks. It was evident that measurement of soft X-ray reflectance properties could have potential as a way of detecting foreign materials in food.

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